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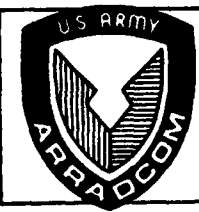
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COMPRESSIBLE FLUIDS INVESTIGATION

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LARGE CALIBER
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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*
This report describes an investigation of compressible fluids for use in artillery recoil mechanisms. An examination of chemical theory revealed the ideal properties which a compressible fluid should possess. More than 20 sample fluids were evaluated in a series of hydrostatic and miscibility tests. Results indicate that perfluorinated compounds exhibit better qualities for artillery applications than the silicone oil currently in use.
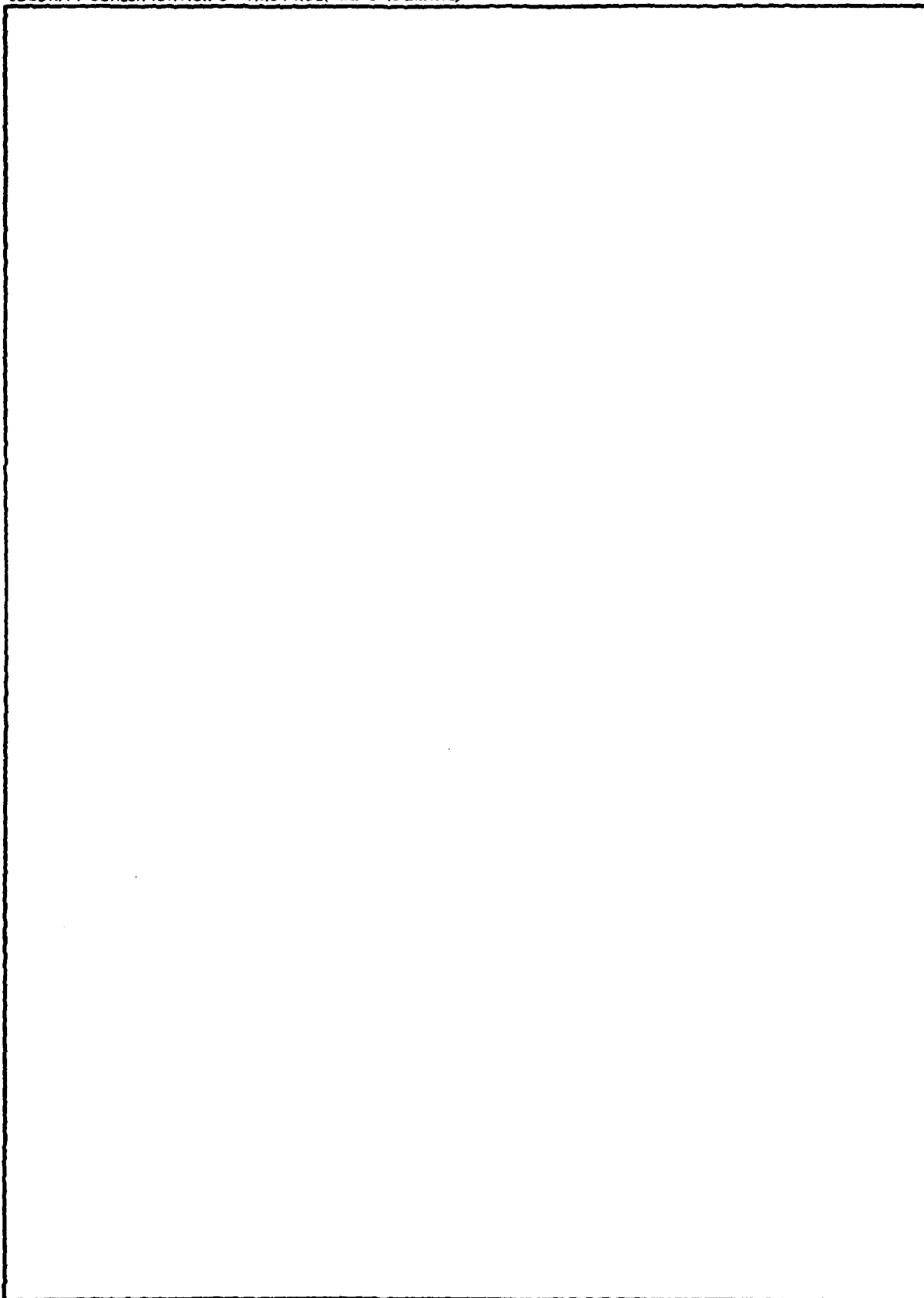
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1.0 INTRODUCTION

For maximum efficiency of the compressible fluid recoil mechanism, a fluid with as high compressibility as possible is needed, particularly at low temperatures. At present, DC-200 (10) (a Dow Corning silicone fluid with a viscosity of 10 cs at room temperature) is being used in testing the prototype and in the compressible fluid test fixture (CFTF). Results of a previous ARRADCOM program (ref 1) indicated that some molecular structures could provide increased compressibility. A recommendation was made to survey the availability of other fluids and to evaluate them for compressibility. The current program was conceived as both a study of the relationship between molecular structure and compressibility and as a further survey of commercially available fluids. As such, this program was an extension of the initial ARRADCOM effort.

In the current program advantage was taken of an increased interest in compressible fluids, studies of liquid theory, and use of "perfluorinated" liquids for medical, hydraulic, and electronic applications. The use of "liquid springs," which depend on both the deformation of a cylinder wall and the compressibility of a hydraulic fluid for a "spring" effect, is at least partially responsible for the current interest in liquid compressibility. A number of papers have been published recently on the effect of pressure on a variety of liquids. Fluorinated compounds with unique structures have been synthesized for use as blood substitutes. A number of companies are offering liquids of various structures for hydraulic use (fluorinated and perfluorinated ethers, polysiloxanes, polyphosphates and polysilicates, hydrocarbons) and for electronic use (perfluorocarbons and polysilicates). Compounds for further evaluation in this program were selected on the basis of previous test data, current literature, and commercial availability.

2.0 SUMMARY AND CONCLUSIONS

A pressure/volume/temperature (PVT) device was constructed for testing the compressibility[1] of fluids under static conditions. The compressibilities of a series of nine fluorinated and perfluorinated polyethers, seven polysiloxanes and silanes, and ten special compounds of known chemical structure were measured at -46, 25, and 66°C in increments of 1000 psig up to 5000 psig. The results were analyzed statistically to describe the data mathematically and to estimate test error ($\tilde{\sigma}$ = ‹ 0.2%).

Five commercial fluids were found to be more compressible than DC-200 (10) within the test temperature range. A low molecular weight[2] fluorinated polyether exhibited a compressibility at 5000 psig of 3.4 versus 2.6 for DC-200 (10) at -46°C.

If molecular weight is used as a criterion, the fluorine compounds exhibit higher compressibilities than other general structures either tested on this program or for which data were available in the literature. However, if molecular volume is used as a criterion, the siloxane structure apparently affords greater compressibility at temperatures above -46°C. A possible explanation is that the behavior of the fluorine compounds is due to the weak intermolecular association of the "shielding" fluorine atoms whereas the siloxane behavior is due to the wider angle of the silicon-oxygen bond as contrasted to the carbon-oxygen bond.

A limited study of binary systems did not provide a practical fluid with high compressibility. Two observations are noteworthy; isopentyl ether is a good solvent for perfluorinated compounds and heptane/1,5-heptadiene exhibits a nonideality of solution.

Appendix A contains an outline sketch of the test apparatus. Appendix B contains a material safety data sheet for perfluorinated hydraulic fluid.

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1. As used herein, compressibility is defined as the percent change in volume ($\Delta V/V_0$ X 100) at a specified pressure and temperature, where V_0 = volume at ambient pressure.

2. Molecular volume is defined as the volume of one gram mole (specific volume X molecular weight).

3.0 RECOMMENDATIONS

A fluorinated polyether (FE-2) is tentatively recommended for low temperature application. For use in the higher temperature range, a higher molecular weight oligomer (FE-4) or the perfluorinated polyethers (Fomblin D-1 or Brayco 814-Z) are tentatively recommended. However, further testing under dynamic conditions, further characterization of the fluids, and trade-off studies are necessary.

The following tasks are recommended as a follow-on to the current effort:

- Supplement the data available from the vendors and in the open literature to characterize the candidate fluid or fluids to be tested in the CFTF (lubricity, compatibility, air solubility, shear stability, etc.).

- Explore methods of cost reduction/improvement and conduct trade-off studies on the primary candidate fluid(s) considering modifications of molecular structure, method of synthesis, formulation, and desirable properties.

- Modify candidate fluid(s) by formulating (incorporation of anti-oxidants, anti-corrosion agents, stabilizers, etc.) as necessary to improve their operational properties.

4.0 TECHNICAL DISCUSSION

This section is comprised of discussions of theory and approaches, description of the experiments conducted, analysis of the data, and rationale for the recommended fluids

4.1 THEORY AND APPROACHES

According to the concept of continuity of states, liquids can be viewed as a transitional phase having some properties of both solids and gases. The transition can occur over a temperature range of several hundred degrees centigrade from the melting point (slightly dependent on pressure), where solid and liquid can exist in equilibrium, to the critical temperature, where the substance can no longer exist as a liquid regardless of the pressure. At any temperature within this range, applied external pressure will have the effect of restricting molecular motion, close-packing the molecules and, therefore, increasing intermolecular repulsion. This is supported by the observed effect of temperature; if increased, the density and viscosity will decrease, and vice versa. Intermolecular repulsion is apparent from the increased pressure with increased temperature at constant volume. Within certain constraints, the PVT relationship can be extended to liquids.

Since liquids were shown to be slightly compressible, many attempts have been made to describe the phenomenon mathematically and physically. Excellent reviews of proposed theories and equations of state can be found in the literature. (References 2 and 3 are suggested.) Theories attempt to explain compressibility of a liquid in terms of "holes", "cells" or by interaction between molecules. In the case of nonideality of solutions, two basically different theories have been proposed; those based on physical intermolecular forces, and those based on chemical interaction between molecules. Prediction of compressibility has not been successful in either case and even the more recently proposed equations of state (refs 4 and 5) require experimentally derived coefficients.

The approach taken in this program consisted of two phases:

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1) Construction of a simple PVT apparatus, similar in most respects to that used in the previous ARRADCOM program (ref 1), for measuring the compressibility of test liquids directly, and

2) a limited investigation of the associative properties of binary solutions.

4.2 SELECTION OF FLUIDS

There were three criteria in the selection of single fluids to be tested: (1) test data in the literature and from the previous ARRADCOM investigation (ref 1); (2) suitability of compounds based on general operational requirements; and (3) compounds having potentials for furthering the understanding of molecular structure and compressibility relationships.

Because one of the major objectives of the program was to improve compressibility at low temperatures, a number of compounds were excluded as candidates. Only sparse data were available at low temperatures; however, it was assumed that relatively low values at room temperature signified poor compressibility at low temperatures as well. Phosphate and silicate esters were eliminated on the basis of high bulk modulus data reported on two such fluids by Chevron (ref 6). Polyvinylacetates, polyethylene, polyvinylchlorides, polymethacrylates, and polyisobutylenes exhibited low compressibilities at room and elevated temperatures (ref 7). The compressibilities of polybutadiene, styrene, and their copolymers (ref 8) were also too low for consideration. Materials with functional groups conducive to hydrogen bonding, such as hydroxyl groups, were excluded because these would be highly associated and would be expected to exhibit high viscosities and poor compressibilities. Chlorine-containing materials were excluded because they apparently offered no advantage in compressibility (ref 1) over fluorine compounds and were considered potentially more corrosive.

Prime candidates for further characterization were the fluorinated ethers, fluorocarbons, and polysiloxanes. Reported advantages of the fluorine compounds, particularly the fluorinated polyethers, are stability

at high temperatures, chemical inertness, good lubricating quality, and non-flammability. The siloxanes are somewhat inferior in some respects, but have the advantage of low viscosity coefficients. Two of the fluids tested [DC-200 (10), Fomblin YO-4] had been tested in the previous ARRADCOM program (ref 1). However, retesting was necessary for data comparison and to aid in the evaluation of molecular weight/compressibility relationships, which will be discussed in more detail in a subsequent section.

A number of compounds were tested to assess the effects of chemical structure on compressibility. These were tetra-alkylsilanes, "block" compounds consisting of both perfluoro and hydrocarbon moities (ref 9), and perfluorocarbons.

The fluids tested, their chemical names, structures, and source are presented on table 1. Their molecular weights (or approximated values), boiling points, critical temperatures (where available), pour points, and densities and viscosities at -46, 25, 66°C are listed in tables II, III and IV.

4.3 DESCRIPTION OF THE COMPRESSIBILITY TEST APPARATUS

Figure 1 is a schematic of the apparatus constructed for measuring liquid compressibility. Essential features of the design are (1) vertical orientation of all components (except for a line pressure transducer) to minimize possibilities of entrapped air; (2) separation of pump and test fluid by a floating piston; (3) capability of testing as little as 25 ccs of liquid; (4) a precision of ± 0.05% in volume change measurements; (5) provision of a degassing chamber to permit degassing of test fluid after filling the apparatus; and (6) absence of absorbent seals within the valves and pressure test chamber. The entire apparatus was enclosed in a temperature-controlled cabinet equipped with a circulating fan. Liquid nitrogen was used as the coolant; heating coils were used to attain elevated temperatures. The correction for volume expansion on internal compression at 5000 psig was .0035 cc, an order of magnitude less than the precision of measuring the volumetric change from the calibrated expansion tube. The volume of the test fluid compression chamber was 6.50 ccs.

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Table I. Abreviations, Chemical Names, Structures, and Vendors of Tested Fluids

Fluid	Chemical Name	Structure	Source
FE-2	Poly-1,2-epoxy hexa-fluoropropane ether	$F{-}\left(CF{-}CF_2O\right)_n{-}CFCF_3$ with CF_3 branch, H ; $n = 2$	Helix Associates, Newark, DE
FE-4	Same as above	Same as above $n = 4$	Same as above
D-1	Polyperfluoroisopropyl ether ("Fomblin Y" Fluids)	$\left[{-}O{-}CF{-}CF_2{-}O{-}CF{-}CF_2{-}\left(CF_2\right)_x{-}OCF_2{-}\right]_n$ with CF_3 branches	Montedison USA Bridgetown, Mo.
YO-4, Y-25, YVAC 06/6	Same as above	Same as above, higher x and n values	Same as above
3X-823-1, 3X-823-2, 814-Z	Polyperfluoroethyl ethers ("Fomblin Z" Fluids)	$\left[{-}O{-}CF_2CF_2OCF_2CF_2{-}\left(CF_2\right)_x{-}OCF_2{-}\right]_n$	Bray Oil Co. El Monte, Ca.
DC-200 (10) (10 Centistoke)	Polydimethylsiloxane	$H_3C{-}\left(SiO\right)_n{-}Si(CH_3)_3$ with $(CH_3)_2$	Dow Corning Midland, MI.
PS-181	Polymethyl-3,3,3-tri-fluoropropylsiloxane	$(H_3C)_3\,SiO{-}\left(\begin{array}{c}CH_3\\ SiO\\ CH_2\\ CH_2\\ CF_3\end{array}\right)_n{-}Si(CH_3)_3$	Petrach Systems, Inc. Bristol, PA.

Table I. Abreviations, Chemical Names, Structures, and Vendors of Tested Fluids (Cont'd.)

Fluid	Chemical Name	Structure	Source
PS-061	Polymethyl (8-12%) phenyl-methyl siloxane copolymer	$H_3C \left(\begin{array}{c} C_6H_5 \\ SiO \\ CH_3 \end{array} \right)_x \left(\begin{array}{c} CH_3 \\ SiO \\ CH_3 \end{array} \right)_y Si(CH_3)_3$	Same as above
S-1174 (Mod)	3-(heptafluoroisopropoxy) - propyl - di-(trimethyl) siloxy) silane	$(CF_3)_2CFO(CH_2)_3 Si\left[O \underset{CH_3}{Si} (CH_3)_3\right]_2$	Silar Laboratories, Inc. Scotia, NY
DBMES	Dibutylethylmethyl silane	$\left[CH_3(CH_2)_3\right]_2 Si \overset{CH_3}{\underset{C_2H_5}{<}}$	Same as above
TBMS	Tributylmethylsilane	$\left[CH_3(CH_2)_3\right]_3 Si\ CH_3$	Same as above
TBS	Tetrabutylsilane	$\left[CH_3(CH_2)_3\right]_4 Si$	Petrach Systems, Inc. Bristol, PA.
H/1-5 HD	Mixture of n-heptane and 1,5-heptadiene	C_7H_{16} and $CH_2 = CH(CH_2)_2 CH=CH\ CH_3$	Chemical Procurement Laboratories, College Point, N.Y.
PFMD	Perfluoro-1-methyl decalin		PCR Research Chemicals, Inc. Gainesville, Fla.

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Table I. Abreviations, Chemical Names, Structures, and Vendors of Tested Fluids (Cont'd.)

Fluid	Chemical Name	Structure	Source
FC-72	Perfluorohexane	C_6F_{14} (Mixed isomers)	Commercial Chemicals Div/3M St. Paul, MN ("Fluorinert" Liquids)
FC-104	Perfluorooctane	C_8F_{18} (Mixed isomers)	Same as above
FC-80	Perfluoro-2-butyl tetrahydrofuran	F_2 —— F_2 / F_2 — F—— C_4F_9 / O (Plus isomers)	Same as above
FC-77	Mixture of perfluorooctane and above compound		Same as above
KS-1	Ethylperfluorodimethyl-perfluoropropylmethane	$C_3F_7 \cdot C(CF_3)(CF_3) - R$ ($R = C_2H_5$)	Dr. Kirby Scherer, consultant
KS-2	Methylperfluorodimethyl-perfluoropropylmethane	Same as above, ($R = CH_3$)	Same as above
KS-3	Isobutylperfluorodimethyl-perfluoropropylmethane	Same as above, $R = CH_2CH(CH_3)_2$	Same as above

Table II. Physical Properties of Fluorinated and Perfluorinated Polyethers[1]

Fluid	M.W.,g.	Tb,°C	Tc,°C	Pour Point,°C	Density,g/cc, at°C			Viscosity, cs, at °C		
					-46	25	66	-46	25	66
FE-2	452	104	218	-123	1.847	1.660	1.555	5.0	0.8	0.5
FE-4	784	194	295	-94	1.908	1.756	1.665	73	2.5	1.1
D-1	800[2]	215	---	< -30	1.963	1.808	1.724	276	4.8	1.6
YO-4	1500[2]	---	---	< -70	2.039	1.872	1.794	43,000[4]	29[4]	5.7[4]
YVAC-06/6	1950[2]	155/0.1 torr	---	< -50	2.016	1.877	1.792	>10,000[4]	50[4]	10.5[4]
Y-25	3000[2]	---	---	< -30	2.055	1.877	1.791	>100,000	170[4]	22[4]
3X-823-1	1800[3]	150/8 millitorr	---	< -68	1.965	1.812	1.720	123	10	2.0
3X-823-2	2600[3]	250/8 millitorr	---	< -68	2.007	1.822	1.721	375[5]	24	5
814-Z	4000[3]	---	---	< -90	1.950	1.814	1.729	∿500[5]	30	9

(1) Molecular weights (M.W.), boiling points (Tb), critical temperatures (Tc), and pour points are vendor data. Density and viscosity data are TRW data, except where otherwise noted.
(2) Average molecular weights.
(3) Average molecular weights, estimated by analogy with other Mortedison (Y series) fluids.
(4) Vendor viscosity data.
(5) Estimated by extrapolation.

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Table III. Physical Properties of Siloxanes and Silanes[1]

Fluid	M.W.,g.	Tb, °C	Pour Point,°C	Density,g/cc, at°C			Viscosity, cs, at°C		
				-46	25	66	-46	25	66
DC-200(10)	980	---	-100	0.999	0.935	0.898	100[2]	21[2]	14[2]
PS-181	1500-3500	---	<- 40	1.344	1.245	1.194	---	295	59
PS-061	1550	---	<- 55	1.047	0.985	0.945	---	53	24
S-1174(Moa)	448	65-68/1 torr	<- 50	1.157	1.079	1.031	39	2.7	1.3
DBEMS	186	40-42/1 torr	---	0.833	0.798	0.767	38[3]	2.0	1.1
TBMS	214	55/1 torr	---	0.833	0.807	0.782	---	3.5	1.3
TBS	256	230-232	---	0.920	0.856	0.829	79	2.6	1.2
PS-140	---	---	---	0.955	0.904	0.867	---	600	161

(1) Molecular weights (M.W.), boiling points (Tb), and pour points are vendor data or estimated from vendor data.
(2) Vendor viscosity data.
(3) Measured at -49°C.

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Table IV. Physical Properties of Special Compounds for Structural Studies

Fluid	M.W.,g.	Tb,°C[1]	Pour Point,°C[1]	Density,g/cc, at °C			Viscosity,cs, at°C		
				-46	25	66	-46	25	66
Heptane	100	98.5	---	---	0.682	---	---	---	---
H/1-5HO	---	---	---	---	0.676	---	---	---	---
PFMD	493	159-160	-70	2.132[1]	1.972[1]	1.877[1]	122	3.2	1.2
FC-72	340	56	-90	1.855[1]	1.665[1]	---	1.4[1]	0.41[1]	---
FC-104	435	101	-65	1.927[1]	1.747[1]	1.643[1]	4.6[1]	0.75[1]	0.41[1]
FC-77	415	97	-110	1.947[1]	1.773[1]	1.672[1]	5[1]	0.8[1]	0.43[1]
FC-80	420	102[2]	-80[2]	1.898[2]	1.757[2]	1.658[2]	5.3[2]	0.8[2]	0.44[2]
KS-1	342	103	---	1.729	1.623	1.540	6.1	0.88	0.58
KS-2	334	85	---	1.822	1.663	1.563	4.6	0.85	0.55
KS-3	376	132	---	1.639	1.506	1.428	9.9	1.1	0.66

(1) Vendor data.
(2) Data by analogy with 3M Fluid FC-75.

TO VACUUM PUMP

DEGASSING CHAMBER

CALIBRATED VOLUME
EXPANSION TUBE

TEST FLUID MENISCUS

VALVE OPENED AT STATIC
PRESSURE TO READ Δ VOLUME

COMPRESSION CHAMBER
(TEST FLUID)

FILLER TUBE

VALVE CLOSED AT DESIRED
STATIC PRESSURE

PRESSURE TRANSDUCER

FILLER VALVE

TEST FLUID

HYDRAULIC PISTON/CYLINDER
ASSEMBLY

MECHANICAL COUPLE

DRIVING HYDRAULIC PISTON/
CYLINDER ASSEMBLY
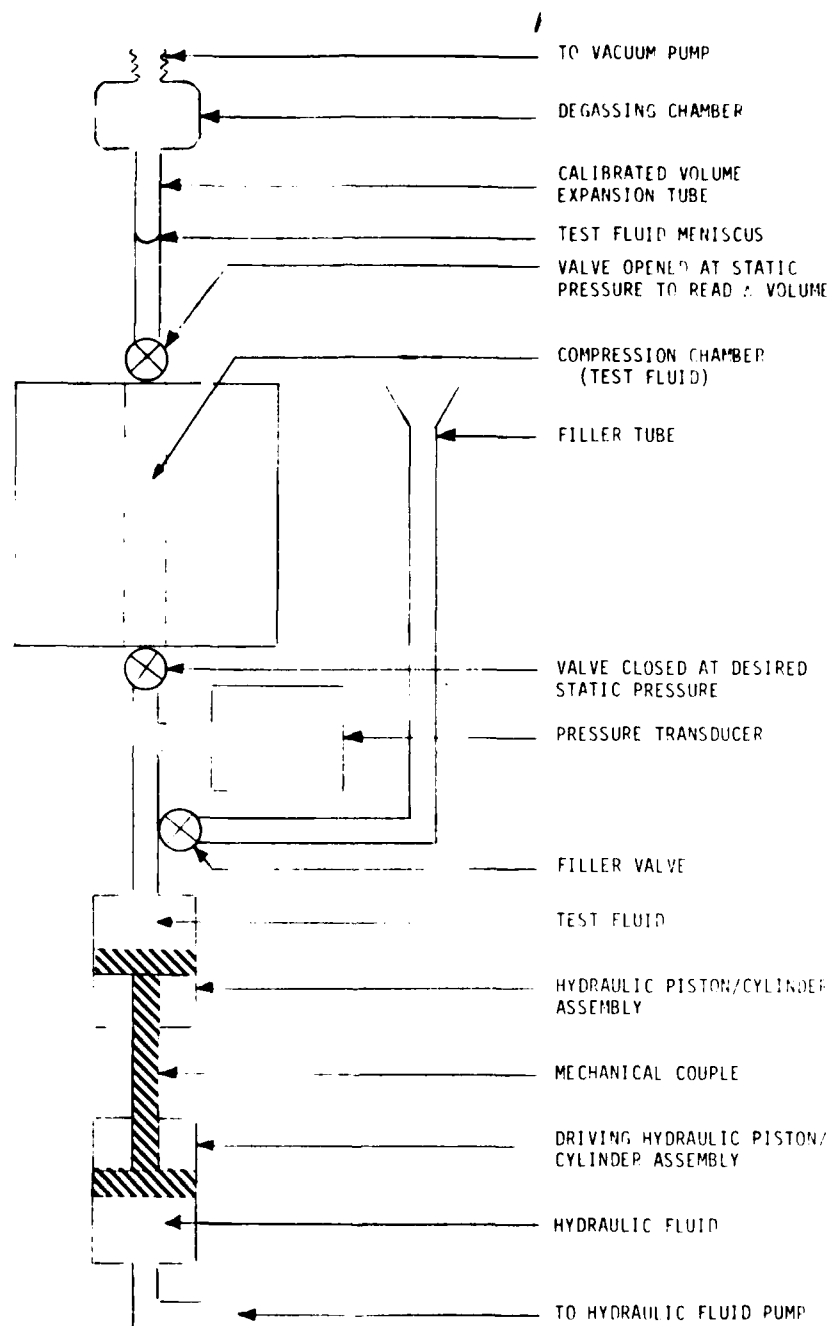
HYDRAULIC FLUID

TO HYDRAULIC FLUID PUMP

Figure 1.   Schematic of the PVT Test Apparatus

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The test fluid was introduced via the filler cup and tube with the compression piston in the upper part of the cylinder. A slightly reduced pressure was used to facilitate flow of the more viscous fluids. With the filler valve closed, the piston was slowly withdrawn to the bottom position. After degassing under reduced pressure, the fluid was raised to a convenient level in the calibrated expansion tube. With the upper valve closed, the fluid was compressed to the desired pressure. The bottom valve was closed and the meniscus in the calibrated tube recorded. The upper valve was opened slowly, and the new meniscus level recorded. The compression/decompression cycle was repeated three times for each pressure level (increments of 1000 psig to 5000 psig). The sigma of the three readings was nominally 0.002 ccs.

4.4 DATA ANALYSIS

One of the principal sources of error in such a device is trapped or entrained air (refs 10 and 11). Although the test fluid was degassed whenever the boiling point was high enough to permit it, there still remained the possibility of air pockets or air bubbles clinging to the surfaces within the compression chamber. Since a plot of the compressibility versus pressure must intercept at the origin, any displacement from the origin can be attributed reasonably to compression of the air bubbles. A shift of the plotted data to the origin is, therefore, a convenient method of correcting for errors due to trapped or entrained air.

In order to develop a consistent method of correction by shifting the curve, and to improve the definition of the compressibility values, the data were analyzed statistically. A least squares computer program was utilized to determine the best fit, using seven mathematical models. The second degree polynomial was found to best represent the data. The curve for each experimental set of data were shifted by imposing a zero intercept ($y = 0 + b_1 x + b_2 x^2$). In most cases (60 out of 75 sets of data) the shift in compressibility was 0.2% or less. The constants for each of the fluids are listed in tables V, VI, and VII.

A typical uncorrected curve of % compressibility versus pressure is shown in figure 2. The three points shown as the 'first experiment' were obtained in the first experiment with the test apparatus; the 'second

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Table V. Fluorinated and Perfluorinated Polyethers,
Statistical Analysis of Compressibility Data

Fluid	Temp,°C	$b_1^{(1)}$(x 10^3)	$b_2^{(1)}$(x 10^8)	Compressibility @ 5000 psi, %
FE-2	-46	0.858	-3.61	3.39
	23	1.363	-5.381	5.47
	66	1.976	-10.722	7.19
FE-4	-46	0.666	-2.00	2.80
	23	1.15	-4.93	4.54
	66	1.36	-4.21	5.77
D-1	-46	0.741	-3.29	2.88
	23	0.882	-0.464	4.30
	66	1.36	-3.64	5.88
YO-4	-46	0.705	-2.93	2.79
	23	0.985	-3.93	3.94
	66	1.12	-1.57	5.19
YVAC 06/6	23	0.904	-1.14	4.23
	66	1.05	1.11	5.56
Y-25	23	0.616	1.00	3.33
	66	0.895	-1.29	4.15
3X-823-1	-46	0.587	-1.29	2.61
	23	0.965	-2.50	4.20
	66	1.49	-6.93	5.73
3X-823-2	-46	0.601	-0.286	2.83
	23	1.01	-2.64	4.41
	66	1.06	-1.21	5.00
814-Z	-46	0.460	2.22	2.85
	23	0.867	-0.857	4.12
	66	1.30	-3.21	5.69

(1)Second degree polynomial constants for $y = 0 + b_1 x + b_2 x^2$

Table VI. Siloxanes and Silanes, Statistical Analysis of Compressibility Data

Fluid	Temp, °C	b_1 $(\times 10^3)$ [1]	b_2 $(\times 10^8)$ [1]	Compressibility @ 5000 psi,
DC-200	-46	.384	2.59	2.57
	23	.920	-2.36	4.01
	66	1.19	-4.71	4.80
PS-181	-30	.356	5.79	3.33 [2]
	23	.643	-1.29	2.89
	66	.744	.357	3.81
PS-061	-46	.567	.250	2.90
	23	.774	-1.57	3.48
	66	.950	-.693	4.58
S-1174 (MOD)	-46	.561	-.500	2.68
	23	.958	-2.643	4.13
	66	1.208	-3.786	5.09
DBEMS	-46	.492	-1.79	2.01
	23	.665	-1.07	3.06
	66	.935	-3.46	3.81
TBMS	-46	.469	-.500	2.22
	23	.636	-.429	3.07
	66	.919	-2.50	3.97
TBS	-46	.426	-.571	1.99
	23	.718	-3.00	2.84
	66	.853	-2.29	3.69

[1] Second Degree Polynomial constants for $y = 0 + b_1 x + b_2 x^2$

[2] This value probably in error due to high viscosity.

Table VII. Special Compounds, Statistical Analysis of Compressibility Data

Fluid	Temp,°C	$b_1 (\times 10^3)^{(1)}$	$b_2 (\times 10^8)^{(1)}$	Compressibility @ 5000 psi, %
HEPTANE	-46	0.514	-0.714	2.40
	23	0.761	-0.0714	3.79
	66	1.39	-6.00	5.48
H/1-5 HD	-46	0.387	3.15	2.72
	23	0.770	0	3.85
	66	1.285	-3.286	5.60
PFMD	-46	0.480	-0.643	2.24
	23	0.798	-1.571	3.59
	66	0.779	2.143	4.43
FC-72	-46	0.895	-4.071	3.46
	23	1.379	-3.714	5.97
FC-104	-46	0.613	-0.929	2.98
	23	1.128	-3.786	4.69
	66	1.722	-8.286	6.54
FC-77	-46	0.679	-1.714	2.97
	23	1.024	-1.571	4.73
	66	1.340	-1.071	6.43
FC-80	-46	0.620	-1.429	2.74
	23	0.924	0	4.62
	66	1.597	-7.429	6.13
KS-1	-46	0.481	1.071	2.67
	23	1.138	-5.321	4.26
	66	1.886	-13.79	5.98
KS-2	-46	0.388	1.357	2.28
	23	0.957	-2.179	4.24
	66	1.493	-5.500	6.09
KS-3	-46	0.427	0.929	2.37
	23	0.944	-2.357	4.13
	66	1.287	-5.286	5.11

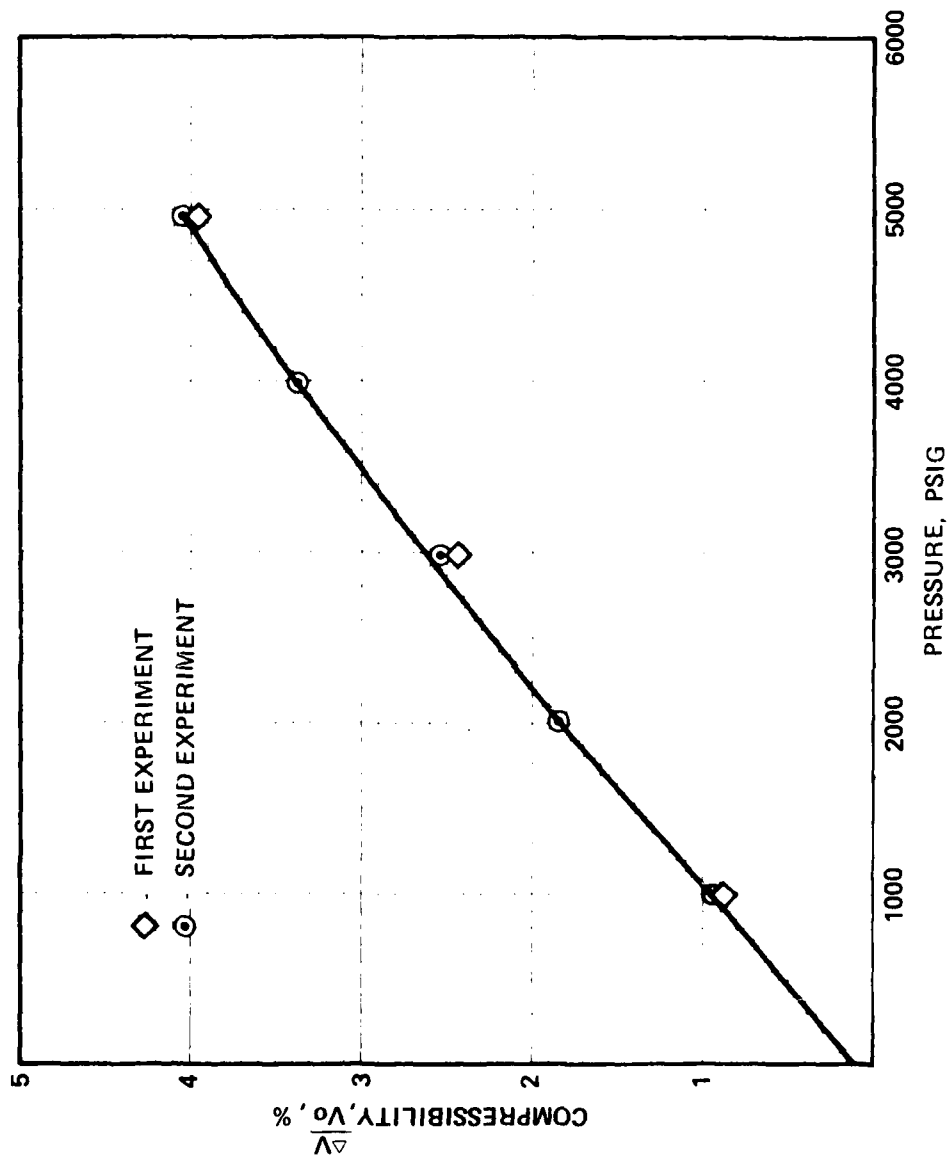[1]Second degree polynomial constants for $y = 0 + b_1 x + b_2 x^2$

Figure 2. Typical Compressibility Versus Pressure Curve

experiment' data were obtained three months later. The data indicate a reasonable repeatability between experiments over the course of the program. Three separate experiments were conducted on the FE-2 fluid for three reasons:

(1) Unusually high intercepts were experienced (>0.2%) in some cases;

(2) The fluid was of particular interest to the program; and

(3) An estimate of sigma (standard error) was desired.

The data at 25°C are shown in figure 3, with superimposed 3-sigma bands. The sigma value, ± 0.2%, is a pooled estimate of standard deviation based on compressibility data at 25 and 66°C.

4.5 DISCUSSION OF RESULTS

The compressibility versus temperature of five commercial fluids are compared to DC-200 (10) in figure 4. All have apparent compressibilities greater than DC-200 (10) over the temperature range of -46 to 66°C (-51 to 151°F). Of particular interest at the lower temperatures is FE-2, which offers a substantial improvement over DC-200 (10). This material is similar to that tested previously (ref 1) (FE-3) but of lower molecular weight. The next higher molecular weight tested on the current program is FE-4, which offers only a (possible) slight advantage over DC-200 at low temperatures but somewhat greater advantage in the higher temperature range [5.8 versus 4.8% for DC-200 (10) at 66°C]. For use in the higher temperature range, D-1 or 814-Z also afford somewhat higher compressibilities. FE-2 and FE-4 are probably most available and lowest in cost, D-1 is considered intermediate, and 814-Z least available and most costly.

The effect of molecular weight (and therefore molecular volume) on compressibility is an important factor in selecting a fluid for use or for further study. Figure 5 is a plot of compressibility of polymethylsiloxanes versus molecular weight extrapolated from data in reference 12. At low molecular weights the effect is dramatic but at higher molecular weights the effect becomes essentially nil. The authors suggest that the independence of molecular weight in the latter region is due to "the maximum number of chemical bonds per unit volume" having been reached.
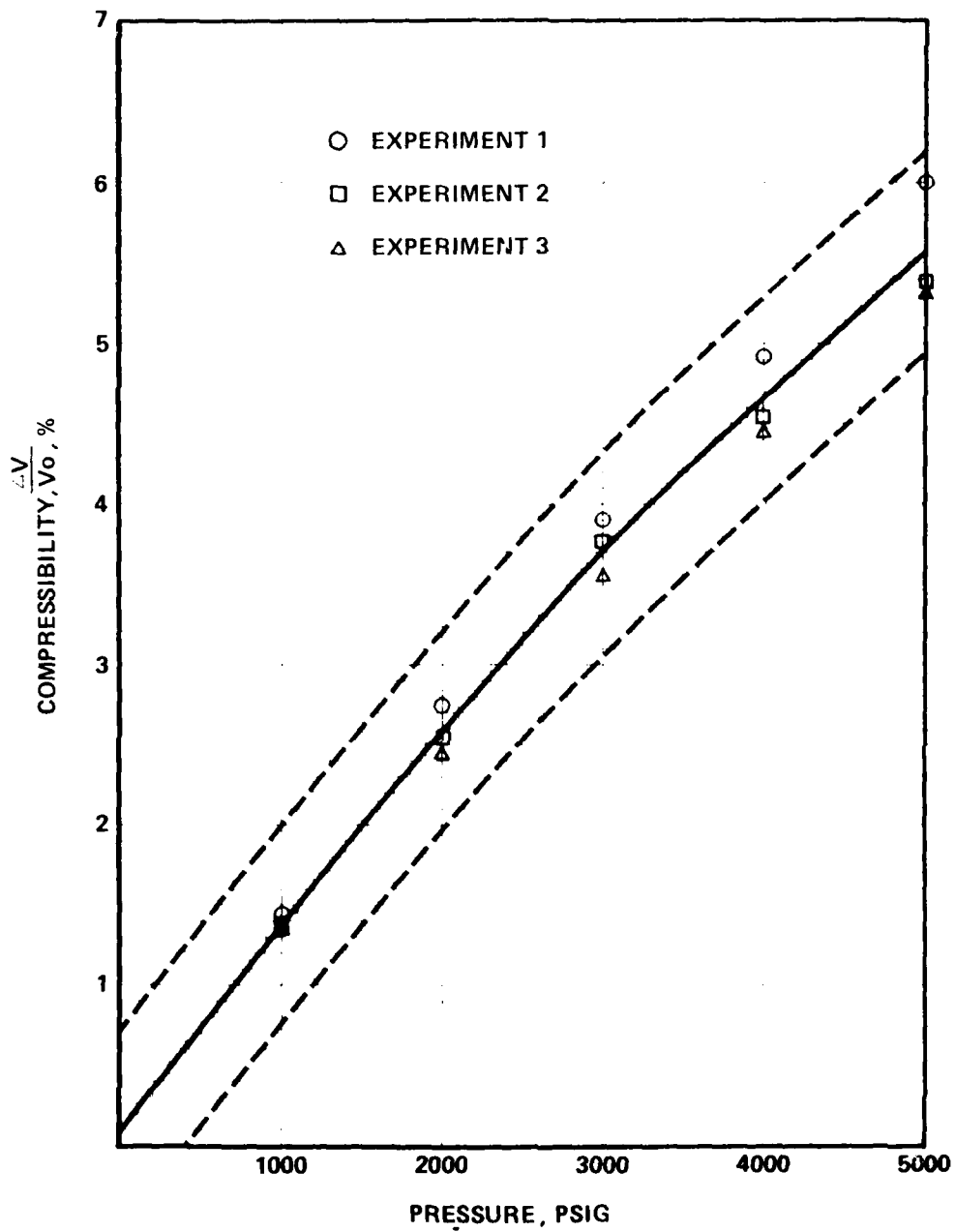
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Figure 3. Compressibility Curve of FE-2 Fluid With Superimposed
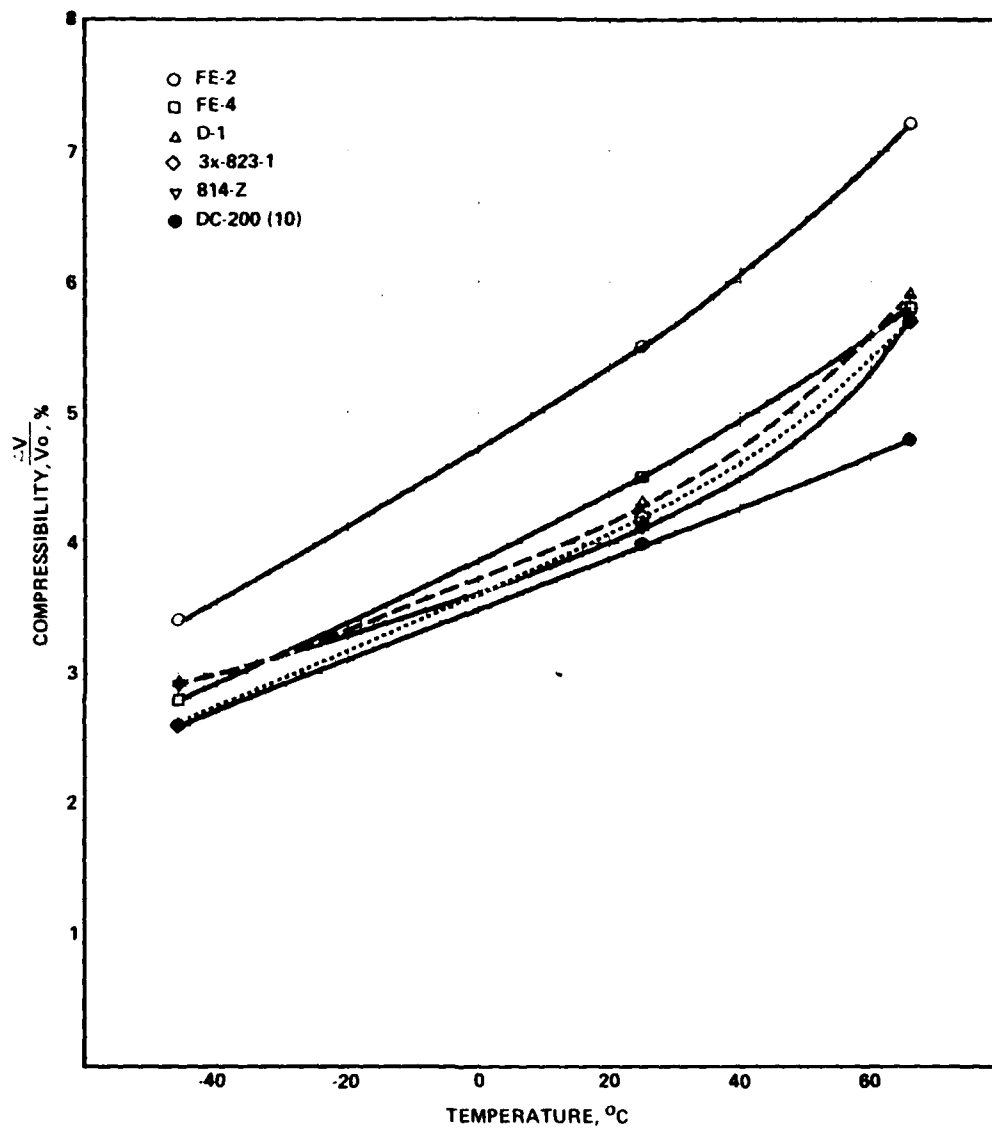3-Sigma Bands

20

Figure 4. Compressibility Versus Temperature of Selected Commercial Fluids at 5000 psig

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Thus for maximum compressibility, the fluid selected should have the lowest possible molecular weight commensurate with other requirements, particularly those at the nigher temperatures.

The molecular weight, specific volume, molecular volume, and compressibility at 5000 psig of the liuids tested on this program are listed in table VIII. It should be noted that the molecular weights of the polymeric materials are only approximate in most cases. However, in each series of compounds with the same chemical structure, the general effect of molecular weight is apparent. For instance, in the fluorinated ether eries, only minor effects are seen at molecular weights over 1000.

The relative effects of molecular weight/compressibility as a function of general molecular structure at 25°C are pictured in figure 6. All compounds are monomeric except the polymethyldisiloxanes and the fluorinated ethers, FE-2 and FE-4. The hydrocarbon values were extrapolated from reference 13. Based on molecular weight alone, the fluorine compounds have higher compressibilities than the others, including the siloxanes.

If compressibilities are compared on a molecular volume basis, the picture is different. Table IX lists the boiling points (Tb), critical temperatures (Tc) and compressibilities at reduced temperatures of compounds having nearly the same molecular volumes (specific volume X molecular weight). If the compressibilities of these compounds are plotted at the actual test temperatures, as shown in figure 7, the disiloxane (HMDS) is superior in compressibility at room temperature and above. At reduced temperatures, as shown in figure 8, all the compounds appear to have nearly the same compressibilities at the lower temperatures and at the higher temperatures except for the disiloxane. It may be noted that HMDS has next to the lowest molecular volume of the compounds listed in table IX. However, the next higher siloxane oligomer (trimer) has a critical temperature of 563°K (ref 14), an estimated molecular volume of approximately 380 cc, and a compressibility of about 4.6% at 25°C and 5000 psig. Even at this high molecular volume, the compressibility of the siloxane appears to be above the population of the other compounds. The greater compressibility of the siloxanes may be related to the flexible silicon-oxygen-silicon bonds which, in turn, are attributed to the greater angle
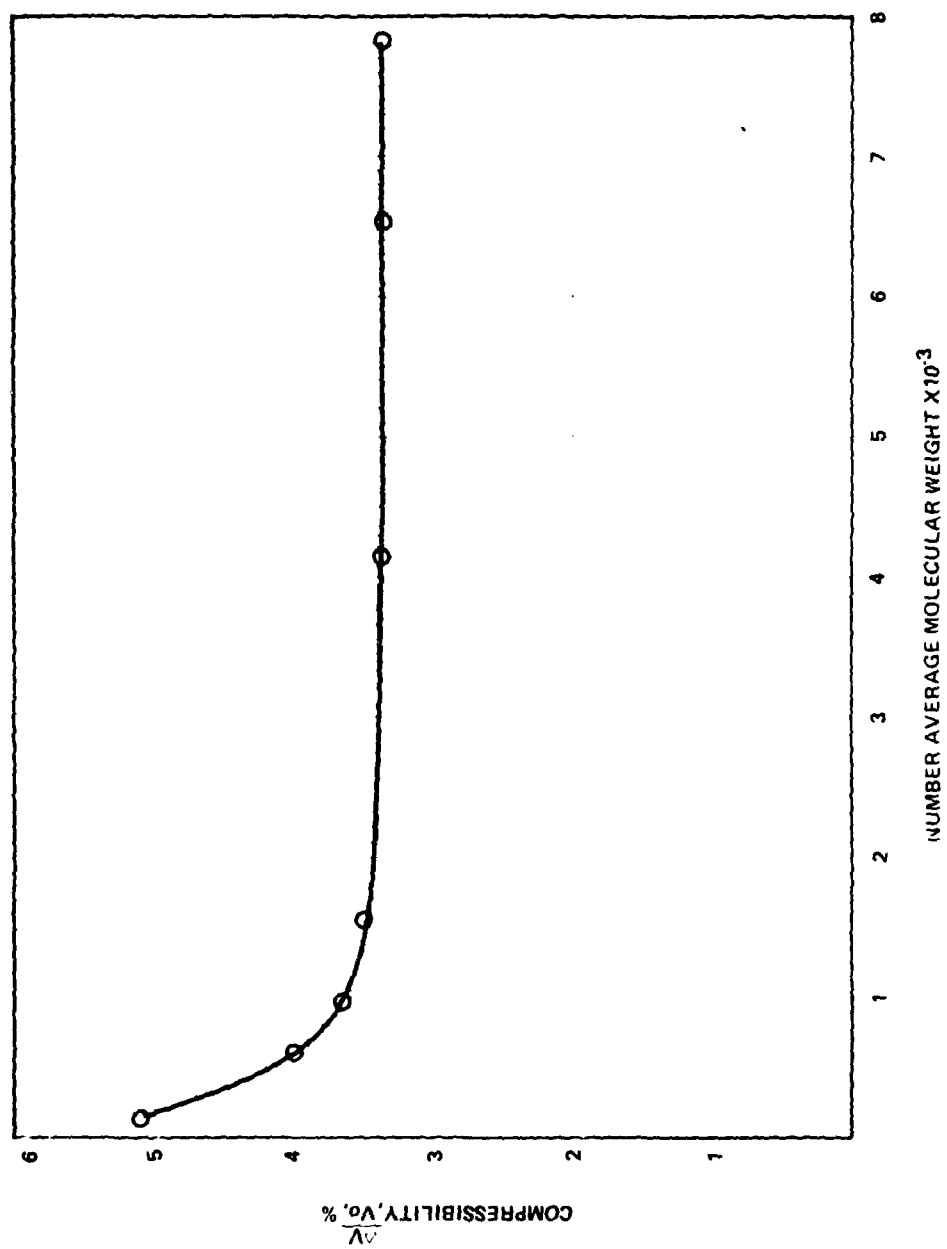
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Figure 5. Effect of Molecular Weight of Dimethylsiloxane Oligomers on Compressibility at 5000 psig and 25°C

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Table VIII. Molecular Weight (M.W.), Molecular Volume, and Compressibility of Tested Fluids

Fluid	M.W.	Specific Volume, cc/g at °C			Molecular Volume, cc, at °C			Compressibility, 5000 psig, % at		
		-46	25	66	-46	25	66	-46	25	66
FE-2	452	.541	.602	.643	245	272	291	3.4	5.5	7.2
FE-4	784	.524	.569	.601	411	446	471	2.8	4.5	5.8
D-1	800	.509	.553	.580	407	442	464	2.9	4.3	5.9
YO-4	1500	.490	.534	.557	736	801	836	2.8	3.9	5.2
YVAC 06/6	1950	.496	.533	.558	966	1039	1088	-	4.2	5.6
Y-25	3000	.487	.532	.558	1460	1598	1675	-	3.3	4.2
.3 X-823-1	1800	.509	.552	.581	916	993	1047	2.6	4.2	5.7
3 X 823-2	2600	.498	.549	.581	1295	1427	1511	2.8	4.4	5.0
814-Z	4000	.513	.551	.478	2051	2205	2313	2.9	4.1	5.7
DC-200 (10)	980	1.001	1.07	1.136	981	1048	1091	2.6	4.0	4.8
PS-181	2500[1]	.744	.803	.838	1860	2008	2094	3.3(-30)	2.9	3.8
PS-061	1500	.955	1.015	1.058	1480	1574	2646	2.9	3.5	4.6
S-1174 (Mod)	448	.864	0.927	.970	387	415	435	2.7	4.1	5.1
DBEMS	186	1.200	1.253	1.304	223	233	243	2.0	3.1	3.8
TBMS	214	1.200	1.239	1.279	257	265	274	2.2	3.1	4.0
TBS	256	1.087	1.168	1.206	278	299	309	2.0	2.8	3.7
PFMD	493	0.469	0.507	0.533	231	250	263	2.2	3.6	4.4
FC-72	340	0.539	0.601	-	183	204	-	3.5	6.0	-
FC-104	435	0.519	0.572	0.609	226	249	265	3.0	4.7	6.5

Table VIII. Molecular Weight (M.W.), Molecular Volume, and Compressibility of Tested Fluids (Con't.)

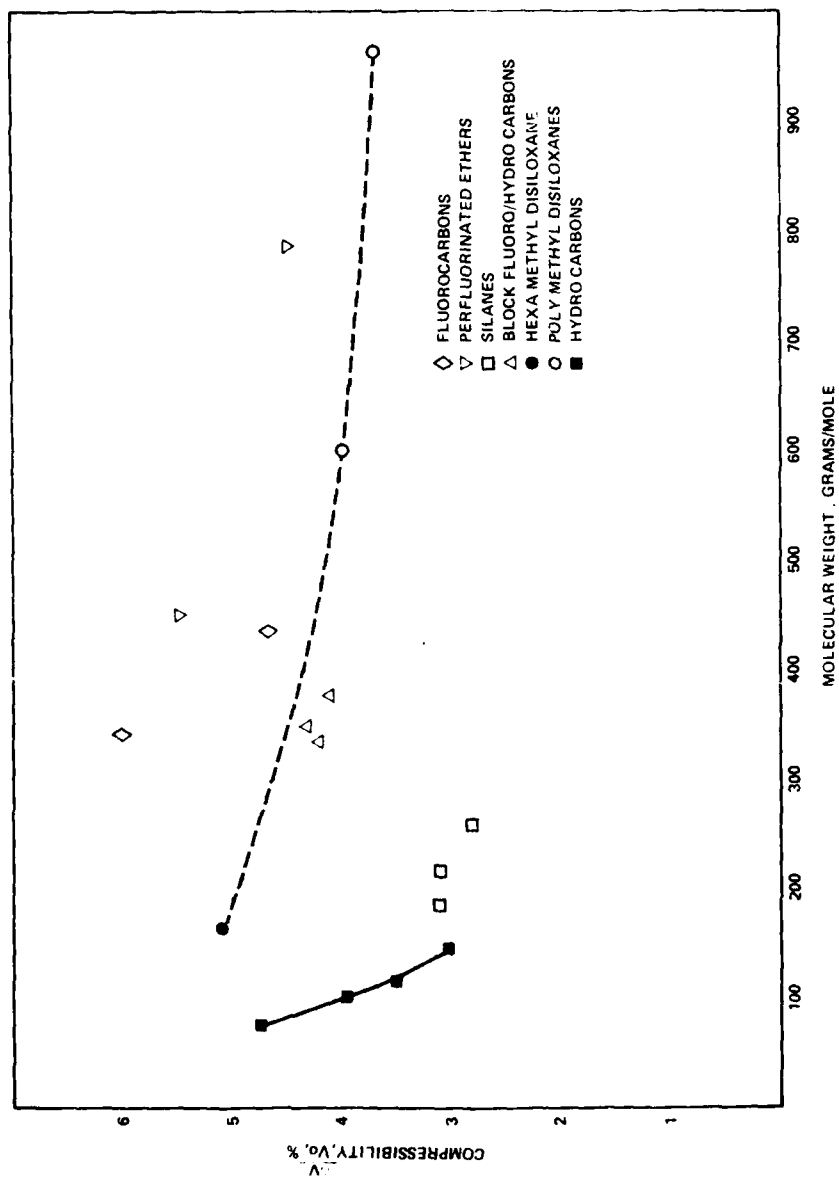| Fluid	M.W.	Specific Volume, cc/g at °C			Molecular Volume, cc, at °C			Compressibility, 5000 psig, % at °C		
		-46	25	66	-46	25	66	-46	25	66
FC-77	415	0.514	0.564	0.598	213	234	248	3.0	4.7	6.4
FC-80	420	0.527	0.569	0.603	221	239	253	2.7	4.6	6.1
KS-1	348	0.578	0.616	0.649	201	214	226	2.3	4.2	6.1
KS-2	334	0.549	0.601	0.640	183	201	214	2.3	4.2	6.1
KS-3	376	0.610	0.664	0.700	229	250	263	2.4	4.1	5.1

(1) Estimated mean value

Figure 6. Relative Effects of Molecular Structure on Compressibility at 5000 psig and 25°C

Table IX. Comparison of Compressibility of Selected Compounds at Reduced Temperatures

Compound	Mole Volume at 25°C, cc	Tb, °K	Tc, °K	Compressibility at 5000psig, %					
				T/Tc [1]	Comp	T/Tc [1]	Comp	T/Tc [1]	Comp
HMDS	213	373	517 [2]	.439	2.9	.576	6.4	.656	12.1
FE-2	272	377	491 [3]	.462	3.4	.607	5.5	.69	7.2
FC-104	249	374	510 [4]	.445	3.0	.584	4.7	.665	6.5
KS-3	250	405	520 [5]	.436	2.4	.573	4.1	.652	5.1
PFMD	250	432	586 [3]	.387	2.2	.508	3.6	.578	4.4
TBMS	265	473	600 [5]	.38	2.2	.50	3.1	.57	4.0
n-Decane	195	447	619 [6]	.367	1.4	.481	2.9	.548	4.0

(1) Test temperature divided by critical temperature

(2) Reference 14

(3) Vendor data

(4) Extrapolated from data in Reference 15

(5) Estimated by method of Lyderson, described in Reference 16
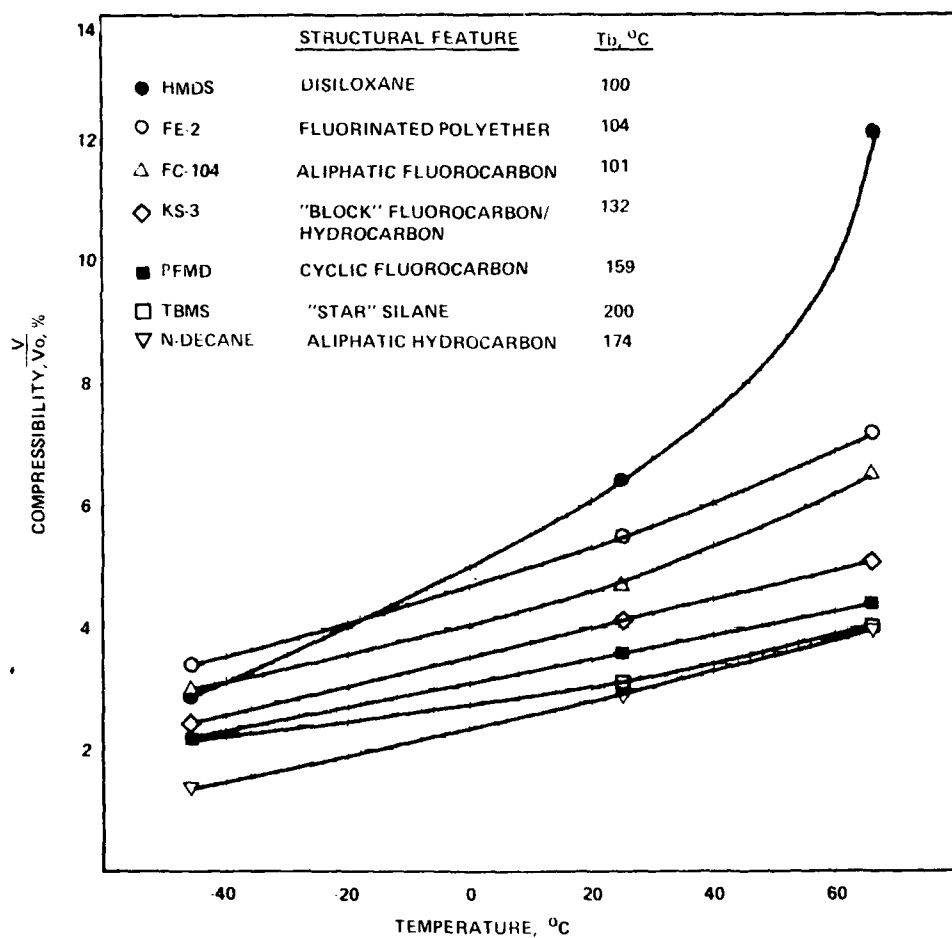
(6) Reference 16

Figure 7.   Compressibility at 5000 psig of Compounds with Molecular
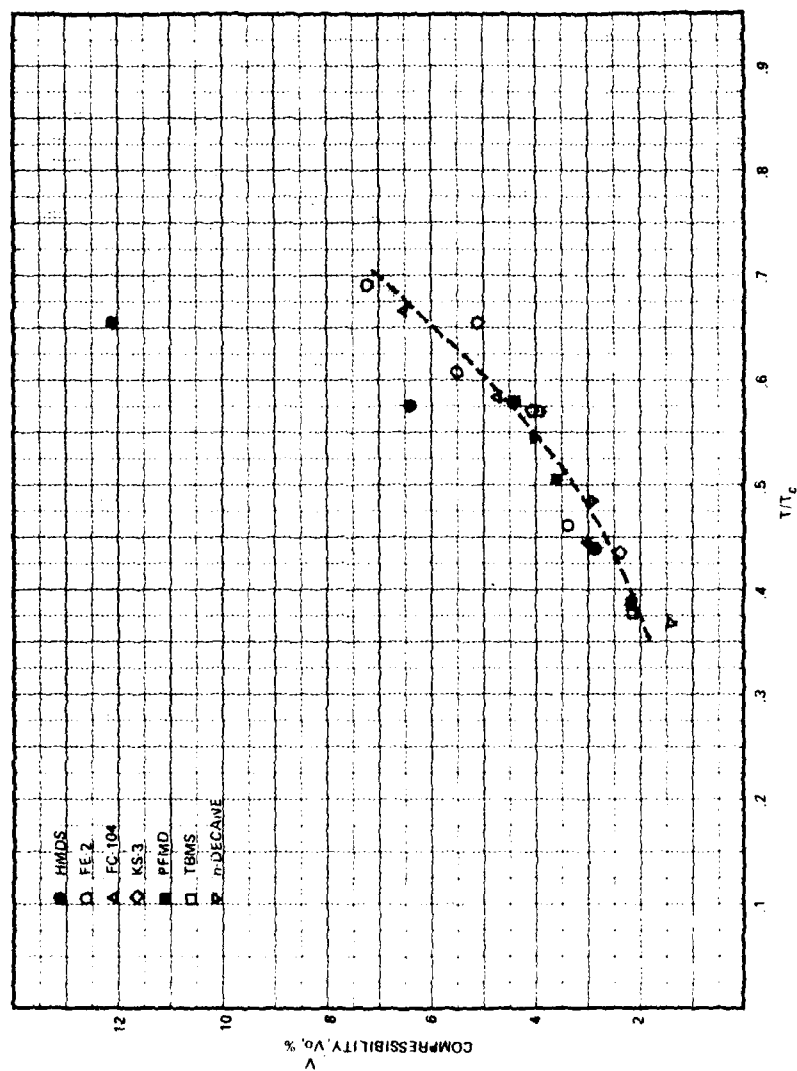Volumes of 195 - 275 cc. as a Function of Test Temperature

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Figure 8.   Compressibility at 5000 psig of Compounds with Molecular Volumes of 195 - 275 cc. at Reduced Temperatures

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of the silicon-oxygen-silicon bond (140-145°) (ref 17) as contrasted to the carbon-oxygen-carbon bond (110°). At the lower temperatures, the fluorine compound's apparently greater compressibility may be due primarily to the weak intermolecular association.

These results are important in the selection of a fluid or fluids because they indicate a need for a better definition of overall requirements and a trade-off study to arrive at an optimized material. As an example, the optimum balance of viscosity and molecular weight has not yet been defined. The fluids studied on this program provide a wide range of viscosities, as shown in figure 9. It would appear that FE-2 is the best selection, considering both compressibility and viscosity and, indeed, this may be true for low temperature application. At higher temperatures, however, a fluid such as 814-Z may be preferred even though a DC-200 fluid may be blended to provide higher compressibility.

4.6 INVESTIGATION OF BINARY SYSTEMS

This approach was based on the chemical theory, originally proposed by Dolezalek, which attributes nonideality of solutions to chemical association and solvation. Accordingly, a compressible fluid should have low chemical reactivity as well as low physical intermolecular attractive forces. As an example, compounds with negligible hydrogen bonding potential, such as hydrocarbons, therefore would be expected to exhibit markedly higher compressibility than those with hydrogen bonding, such as alcohols. Binary systems, or solutions, of interest would be those in which the unlike molecules have a minimal chemical affinity for one another. However, sufficient affinity must be present to attain and maintain solution over the desired temperature range. This would require compositional trade-offs in obtaining optimum solution and compressibility over the desired temperature range.

A series of miscibility experiments were conducted first with various combinations of hydrocarbons, polyesters, perfluorocarbons, siloxanes, and silanes. Some of these have shown promise of synergism in compressibility. Dunlap and Scott (ref 18) found that mixtures of perfluorohexane/hexane had higher compressibilities than either compound alone. However, perfluorocarbon compounds are insoluble in most other liquids below room
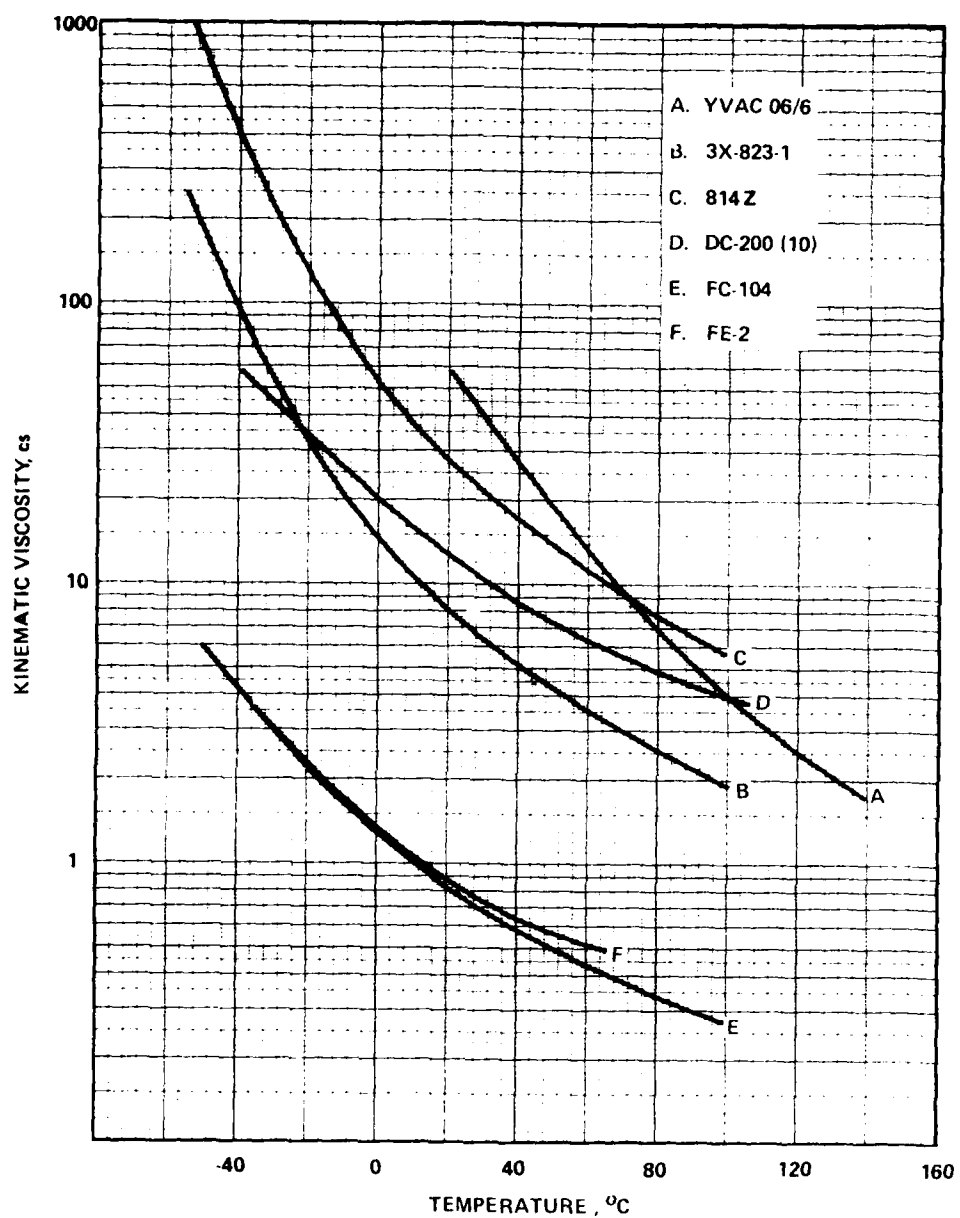
30

Figure 9.   Range of Kinematic Viscosities as Shown by Selected
            Compounds Tested

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temperature. (We found one exception; perfluorotoluene in isopentyl ether. This was not considered a practical liquid and, therefore, compressibility was not measured.) Combinations of polymers, such as DC-200, polybutene, and dioctylphthalate were also found incompatible at -46°C, although mutually soluble at room temperature. Binary systems consisting of poly-isobutylene/benzene or cyclohexane and polydimethylsiloxane/hexamethyl-disiloxane, which would be expected to be compatible at low temperatures, showed no benefit in compressibility in the 25 to 65°C temperature range (ref 19).

Binary systems (1/1 volume ratio) found compatible at low temperatures (-45°C) are listed in table X along with predicted and observed densities. The purpose here was to determine what combination of two chemical structures, if any, would result in a lower-than-predicted density. As shown in the table, the heptane/1,5-heptadiene mixture resulted in a lower density, indicating that this combination would have a higher com-pressibility than either compound by itself. The compressibility data obtained on heptane and on the mixture do indicate a higher value with the latter. However, this would require verification since the increase is small and within the sigma value obtained on FE-2. The value obtained on heptane itself (3.9% at 25°C) agrees well with the value (4.0%) extrapolated from data reported by Bridgman (ref 13).

Three perfluorinated compounds afforded an additional test of a binary system. FC-77 is a mixture of the FC-80 and FC-104 structures. Comparison of the compressibility data tabulated below show no benefit from the mixture:

Temp. °C	Compressibility at 5000 psig, %		
	FC-80	FC-104	FC-77 (Mixture
-46	2.7	3.0	3.0
25	4.6	4.7	4.7
66	6.1	6.5	6.4

Although the binary system approach has been somewhat disappointing to date, some of the results are at least of academic interest. The heptane/1,5-heptadiene mixture, although interesting scientifically, is

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Table X   Binary Systems, Miscible at -45°C

System (1/1 Volume Ratio)		Density, 24°C, g/cc			Density, -45°C	
1	2	Predicted	Measured	Δ, %	Measured	Δ, %
Heptane	2,4-Dimethylpentane	0.67550	0.67629	0.21	0.73480	8.65
	1,5-Heptadiene	0.69821	0.69616	-0.29	0.75202	8.02
	2,4-Dimethyl -2-pentene	0.68422	0.68621	0.29	0.74588	8.7
	n-Heptanol	0.74505	0.75086	0.78	0.80306	6.95
	Tri-n-hexylsilane	0.73527	0.74119	0.81	0.79698	7.53
1,5-Heptadiene	n-Heptanol	0.76229	0.76826	0.78	0.82167	6.95
	Tri-n-hexylsilane	0.75157	0.75716	0.74	0.80721	6.61
DC 200	Tri-n-hexylsilane	0.89331	0.89864	0.60	0.95600	6.38
	Hexane	0.79614	0.80061	0.55	0.85616	6.95
FE-2	Octafluorotoluene	1.6618	1.6708	0.54	1.8260	9.29
	Perfluorohexane	1.6695	1.6739	0.26	1.8194	8.69

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not considered a primary candidate because of the instability and
reactivity of the double bonds, particularly in the presence of oxygen.
The solvent power of isopentyl ether for perfluorinated compounds is
also of interest. Because of the expected low association between
halogens and oxygen, mixtures of such compounds could provide some
advantage.

It should be noted that relatively little information on compressi-
bility of liquid mixtures is available in the literature. Further
understanding of their behavior could still offer advantages. For
instance, there is a possibility for improved performance in blending
polymers with widely different but narrow molecular weight ranges. A
balance of compatibility, intermolecular association, and close-packing
could be advantageous in polymeric compounds other than the siloxanes,
which apparently failed to show any beneficial effect (ref 19).

5.0 CONSIDERATIONS UNDER DYNAMIC CONDITIONS

There are a number of technical questions to be answered in order to firm up the selection of fluids for use under the dynamic processes occurring in a compressible fluid recoil mechanism. Data on many of the desirable properties listed in reference 1 are available from the vendors of commercial liquids such as FE-2, D-1, 814-Z, or DC-200 blends. However, some of the questions cannot be answered adequately without further technical effort. This is discussed briefly below.

The compressibility under dynamic conditions may differ from that at static conditions. This should be verified. The most logical and ready vehicle for such tests is the large scale CFTF at ARRADCOM. Its actual performance can be compared to that predicted by the compressibility values obtained under static conditions. However, smaller scale experiments may be more cost effective and less time consuming. This aspect requires further consideration.

A check on the compatibility of the fluids with the seals and metals in the recoil mechanism is desirable. Although the recommended fluids appear compatible, actual data on specific items are needed to confirm this. If, for instance, the fluorinated liquids plasticize or solvate the polymeric seals to a greater extent than expected, the seals could undergo extrusion and deformation under dynamic conditions. Also the best candidate for low temperature use, FE-2, is not completely fluorinated and the remaining hydrogen may be sufficiently reactive under some conditions to affect the material's reported stability and inertness toward metals.

One of the potentially more troublesome and perhaps least known effect in the use of fluorinated polymers is associated with their unusually high air or oxygen solubilities. Separation of solubilized air under shear and compression/decompression cycling is a problem frequencly encountered in hydraulic systems (ref 10). The mechanism is apparently a decrease in solubility with temperature rise and agglomeration of desolubilized air. In operation, the "bubbles" of air so formed must be compressed before the pressure acts on the liquid itself. The result is a sluggish hydraulic system.

The apparatus constructed for this program may be modified to characterize the fluids with respect to the above phenomenon. After compression/decompression cycling, preferably under adiabatic conditions, the temperature rise, changes in air concentrations, and displacement of the compressibility curve from the zero intercept may be measured. In addition, chemical analyses, such as infrared spectrophotometry, and viscosity measurements may be utilized to check on the thermal and shear stabilities of the fluids.

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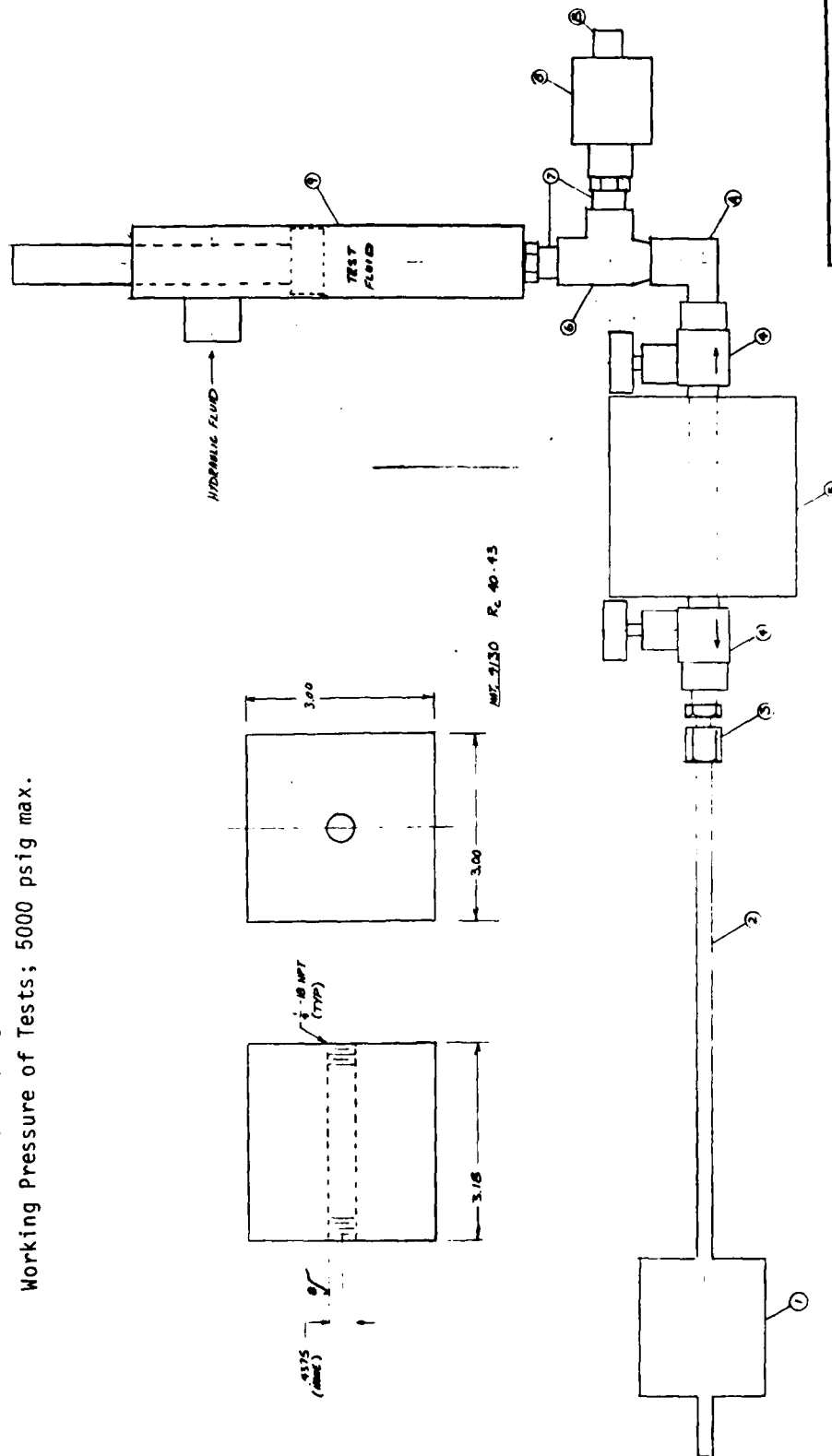
APPENDIX A

DRAWING

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Note: Designed for Working Pressure of 10,000 psig

Tested to 10,000 psig.

Working Pressure of Tests; 5000 psig max.

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APPENDIX B

MATERIAL SAFETY DATA SHEET

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U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

MATERIAL SAFETY DATA SHEET

JAN 16 1981

ENVIRONMENTAL HEALTH

Required under USDL Safety and Health Regulations for Ship Repairing,
Shipbuilding, and Shipbreaking (29 CFR 1915, 1916, 1917)

SECTION I

MANUFACTURER'S NAME	EMERGENCY TELEPHONE NO.
BRAY OIL COMPANY, INC.	(213) 575-1212

ADDRESS (Number, Street, City, State, and ZIP Code)
9550 FLAIR DRIVE, SUITE 301, EL MONTE, CA 91731

MiCRONIC 8147, Hydraulic Fluid

CHEMICAL NAME AND SYNONYMS	TRADE NAME AND SYNONYMS
Perfluorinated Polyalkyl Ether	BRAYCO 814Z

CHEMICAL FAMILY	FORMULA
Perfluorinated Ether	

SECTION II - HAZARDOUS INGREDIENTS

PAINTS, PRESERVATIVES, & SOLVENTS	%	TLV (Units)	ALLOYS AND METALLIC COATINGS	%	TLV (Units)
PIGMENTS			BASE METAL		
CATALYST			ALLOYS		
VEHICLE			METALLIC COATINGS		
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX		
ADDITIVES			OTHERS		
OTHERS					

HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES	%	TLV (Units)
Non-Hazardous as defined by U.S. Dept. of Labor 29CFR, Section 1915.2.		

SECTION III - PHYSICAL DATA

BOILING POINT (°F.)	500	SPECIFIC GRAVITY (H₂O=1)	1.833
VAPOR PRESSURE (mm Hg.)	N/A	PERCENT, VOLATILE BY VOLUME (%)	N/A
VAPOR DENSITY (AIR=1)	N/A	EVAPORATION RATE (=1)	N/A
SOLUBILITY IN WATER	Insol.		

APPEARANCE AND ODOR   Clear, water-white liquid, very mild odor.

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used)	FLAMMABLE LIMITS	Lel	Uel
Non-Flammable, Will not flash.	N/A		

EXTINGUISHING MEDIA   N/A

SPECIAL FIRE FIGHTING PROCEDURES   N/A

UNUSUAL FIRE AND EXPLOSION HAZARDS   N/A

SECTION V - HEALTH HAZARD DATA

Threshold Limit Value
Not determined. Use 5 mg per cubic meter for mist.

Effects of Overexposure
Essentially non-toxic. Not expected to cause any ill effect except possibly in very sensitive individuals.

Emergency and First Aid Procedures
Eye contact - Flush thoroughly with water.

Skin contact - Wipe dry, then wash with soap and water.

Ingestion - Do not induce vomiting, call physician.

SECTION VI - REACTIVITY DATA

STABILITY	UNSTABLE		CONDITIONS TO AVOID	
	STABLE	X	Temperatures above 400°F when in contact with active metals such as aluminum & titanium.	

INCOMPATABILITY (Materials to avoid)
Aluminum chloride and Friedal-Crafts Reagents.

HAZARDOUS DECOMPOSITION PRODUCTS
Contact with active metals above 400°F may induce decomposition to toxic gases.

HAZARDOUS POLYMERIZATION	MAY OCCUR		CONDITIONS TO AVOID
	WILL NOT OCCUR	X	

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED
Mop, wipe up or absorb with clay, diatomaceous earth or other inert material and store in closed metal container.

WASTE DISPOSAL METHOD
Do not burn or contaminate materials that may be burned. Dispose of by method in compliance with local, state and federal regulations regarding health, air and water pollution.

SECTION VIII - SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (Specify type)
None normally required.

VENTILATION	LOCAL EXHAUST None normally required.		SPECIAL
	MECHANICAL (General)		OTHER

PROTECTIVE GLOVES
None normally required.

EYE PROTECTION
Safety glasses recommended.

OTHER PROTECTIVE EQUIPMENT

SECTION IX - SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING
Smokers should wash hands before handling tobacco products. Do not store near active metals that are above 400°F

OTHER PRECAUTIONS
Do not burn anything that has been contaminated with product.

PAGE (2)
GPC 9.0.540

Assistant Secretary of the Army
Research and Development
ATTN: Department for Science and
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